```
=> s dimethyl succinate/cn
L1            1 DIMETHYL SUCCINATE/CN

=> s dimethyl glutarate/cn
L2            1 DIMETHYL GLUTARATE/CN

=> s dimethyl adipate/cn
L3            1 DIMETHYL ADIPATE/CN

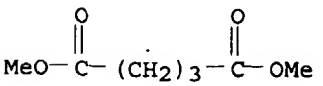
=> s l1 or l2 or l3
L4            3 L1 OR L2 OR L3

=> s 1-3
      14425425 1
      12897299 3
L5     2699485 1-3
                (1(W)3)

=> \end
\END IS NOT A RECOGNIZED COMMAND
The previous command name entered was not recognized by the system.
For a list of commands available to you in the current file, enter
"HELP COMMANDS" at an arrow prompt (=>).

=> d l4 1-3

L4   ANSWER 1 OF 3  REGISTRY  COPYRIGHT 2004 ACS on STN
RN   1119-40-0  REGISTRY
CN   Pentanedioic acid, dimethyl ester (9CI)  (CA INDEX NAME)
OTHER CA INDEX NAMES:
CN   Glutaric acid, dimethyl ester (6CI, 7CI, 8CI)
OTHER NAMES:
CN   DBE 5
CN   Dimethyl 1,3-propanedicarboxylate
CN   Dimethyl glutarate
CN   Dimethyl pentanedioate
CN   Methyl glutarate
CN   NSC 58578
FS   3D CONCORD
DR   111366-62-2
MF   C7 H12 O4
CI   COM
LC   STN Files:   ANABSTR, BEILSTEIN*, BIOBUSINESS, BIOSIS, CA, CANCERLIT,
       CAOLD, CAPLUS, CASREACT, CEN, CHEMCATS, CHEMLIST, CIN, CSCHEM, CSNB,
       DETHERM*, HODOC*, HSDB*, IFICDB, IFIPAT, IFIUDB, MEDLINE, MSDS-OHS,
       NIOSHTIC, PIRA, PROMT, SPECINFO, TOXCENTER, ULIDAT, USPAT2, USPATFULL
         (*File contains numerically searchable property data)
     Other Sources:   DSL**, EINECS**, TSCA**
         (**Enter CHEMLIST File for up-to-date regulatory information)
```



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**PROPERTY DATA AVAILABLE IN THE 'PROP' FORMAT**

             622 REFERENCES IN FILE CA (1907 TO DATE)
              22 REFERENCES TO NON-SPECIFIC DERIVATIVES IN FILE CA
             623 REFERENCES IN FILE CAPLUS (1907 TO DATE)
              24 REFERENCES IN FILE CAOLD (PRIOR TO 1967)

L4   ANSWER 2 OF 3  REGISTRY  COPYRIGHT 2004 ACS on STN
RN   627-93-0  REGISTRY
CN   Hexanedioic acid, dimethyl ester (9CI)  (CA INDEX NAME)
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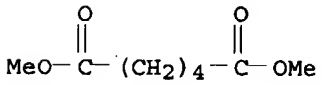
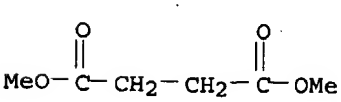
OTHER CA INDEX NAMES:
CN Adipic acid, dimethyl ester (6CI, 8CI)
OTHER NAMES:
CN DBE 6
CN **Dimethyl adipate**
CN Dimethyl hexanedioate
CN Methyl adipate
CN NSC 11213
FS 3D CONCORD
DR 111366-61-1
MF C8 H14 O4
CI COM
LC STN Files: ANABSTR, AQUIRE, BEILSTEIN*, BIOSIS, CA, CANCERLIT, CAOLD, CAPLUS, CASREACT, CBNB, CEN, CHEMCATS, CHEMINFORMRX, CHEMLIST, CIN, CSCHEM, CSNB, DETHERM*, HODOC*, HSDB*, IFICDB, IFIPAT, IFIUDB, MEDLINE, MSDS-OHS, NIOSHTIC, PIRA, PROMT, RTECS*, SPECINFO, TOXCENTER, USPAT2, USPATFULL
(*File contains numerically searchable property data)
Other Sources: DSL**, EINECS**, TSCA**
(**Enter CHEMLIST File for up-to-date regulatory information)



**PROPERTY DATA AVAILABLE IN THE 'PROP' FORMAT**

1138 REFERENCES IN FILE CA (1907 TO DATE)
47 REFERENCES TO NON-SPECIFIC DERIVATIVES IN FILE CA
1140 REFERENCES IN FILE CAPLUS (1907 TO DATE)
49 REFERENCES IN FILE CAOLD (PRIOR TO 1967)

L4 ANSWER 3 OF 3 REGISTRY COPYRIGHT 2004 ACS on STN
RN 106-65-0 REGISTRY
CN Butanedioic acid, dimethyl ester (9CI) (CA INDEX NAME)
OTHER CA INDEX NAMES:
CN Succinic acid, dimethyl ester (6CI, 8CI)
OTHER NAMES:
CN DBE 4
CN Dimethyl butanedioate
CN **Dimethyl succinate**
CN Methyl succinate
CN NSC 52209
FS 3D CONCORD
MF C6 H10 O4
CI COM
LC STN Files: AGRICOLA, ANABSTR, BEILSTEIN*, BIOBUSINESS, BIOSIS, BIOTECHNO, CA, CANCERLIT, CAOLD, CAPLUS, CASREACT, CBNB, CEN, CHEMCATS, CHEMINFORMRX, CHEMLIST, CIN, CSCHEM, CSNB, DDFU, DETHERM*, DIPPR*, DRUGU, EMBASE, GMELIN*, HODOC*, HSDB*, IFICDB, IFIPAT, IFIUDB, MEDLINE, MSDS-OHS, NIOSHTIC, PIRA, PROMT, RTECS*, SPECINFO, SYNTHLINE, TOXCENTER, USPAT2, USPATFULL, VTB
(*File contains numerically searchable property data)
Other Sources: DSL**, EINECS**, TSCA**
(**Enter CHEMLIST File for up-to-date regulatory information)



**PROPERTY DATA AVAILABLE IN THE 'PROP' FORMAT**

1343 REFERENCES IN FILE CA (1907 TO DATE)

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          1344 REFERENCES IN FILE CAPLUS (1907 TO DATE)
            44 REFERENCES IN FILE CAOLD (PRIOR TO 1967)


=> s 1119-40-0/prep
'PREP' IS NOT A VALID FIELD CODE
L6            0 1119-40-0/PREP

=> file caplus
COST IN U.S. DOLLARS                                SINCE FILE      TOTAL
                                                       ENTRY    SESSION
FULL ESTIMATED COST                                    29.14      29.35

FILE 'CAPLUS' ENTERED AT 16:08:26 ON 17 FEB 2004
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FILE COVERS 1907 - 17 Feb 2004  VOL 140 ISS 8
FILE LAST UPDATED: 16 Feb 2004  (20040216/ED)

  This file contains CAS Registry Numbers for easy and accurate
  substance identification.


=> s 1119-40-0/prep
           623 1119-40-0
       3109528 PREP/RL
L7          78 1119-40-0/PREP
                (1119-40-0 (L) PREP/RL)

=> s 627-93-0/prep
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       3109528 PREP/RL
L8         226 627-93-0/PREP
                (627-93-0 (L) PREP/RL)

=> s 106-65-0/prep
          1344 106-65-0
       3109528 PREP/RL
L9         220 106-65-0/PREP
                (106-65-0 (L) PREP/RL)

=> s l7 or l8 l9
MISSING OPERATOR L8 L9
The search profile that was entered contains terms or
nested terms that are not separated by a logical operator.

=> s l7 or l8 or l9
L10        421 L7 OR L8 OR L9

=> s caprolactam and l10
         18908 CAPROLACTAM
L11          4 CAPROLACTAM AND L10

=> s l11 AND WASTE AND ALKALINE
        334409 WASTE
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L12           0 L11 AND WASTE AND ALKALINE

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     3779532 ACID
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               (NITRIC(W)ACID)
L13           1 L11 AND NITRIC ACID

=> s l11 AND NITRIC ACID AND DECOMP?
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     3779532 ACID
       56329 NITRIC ACID
               (NITRIC(W)ACID)
      576273 DECOMP?
L14           1 L11 AND NITRIC ACID AND DECOMP?

=> s l11 AND NITRIC ACID AND DECOMP? AND DISTIL?
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     3779532 ACID
       56329 NITRIC ACID
               (NITRIC(W)ACID)
      576273 DECOMP?
      112407 DISTIL?
L15           1 L11 AND NITRIC ACID AND DECOMP? AND DISTIL?

=> D L11 1-4 IBIB ABS HITSTR

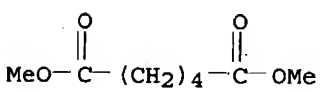
L11  ANSWER 1 OF 4  CAPLUS  COPYRIGHT 2004 ACS on STN
ACCESSION NUMBER:         2003:376877  CAPLUS
DOCUMENT NUMBER:          138:369025
TITLE:                    Preparation of bidentate ligands for use in palladium
                          catalyzed carbonylation of ethylenically or
                          acetylenically unsaturated compounds
INVENTOR(S):              Drent, Eit; Van Ginkel, Roelof; Van der Made, Renata
                          Helena
PATENT ASSIGNEE(S):       Shell Internationale Research Maatschappij BV, Neth.
SOURCE:                   PCT Int. Appl., 23 pp.
                          CODEN: PIXXD2
DOCUMENT TYPE:            Patent
LANGUAGE:                 English
FAMILY ACC. NUM. COUNT:   2
PATENT INFORMATION:

     PATENT NO.        KIND  DATE           APPLICATION NO.   DATE
     ----------------  ----  --------       ---------------   --------
     WO 2003040159      A2   20030515       WO 2002-EP12380   20021105
     WO 2003040159      C1   20030731
     WO 2003040159      A3   20040108
         W:  AE, AG, AL, AM, AT, AU, AZ, BA, BB, BG, BR, BY, BZ, CA, CH, CN,
             CO, CR, CU, CZ, DE, DK, DM, DZ, EC, EE, ES, FI, GB, GD, GE, GH,
             GM, HR, HU, ID, IL, IN, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR,
             LS, LT, LU, LV, MA, MD, MG, MK, MN, MW, MX, MZ, NO, NZ, OM, PH,
             PL, PT, RO, RU, SD, SE, SG, SI, SK, SL, TJ, TM, TN, TR, TT, TZ,
             UA, UG, US, UZ, VN, YU, ZA, ZM, ZW, AM, AZ, BY, KG, KZ, MD, RU,
             TJ, TM
         RW: GH, GM, KE, LS, MW, MZ, SD, SL, SZ, TZ, UG, ZM, ZW, AT, BE, BG,
             CH, CY, CZ, DE, DK, EE, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL,
             PT, SE, SK, TR, BF, BJ, CF, CG, CI, CM, GA, GN, GQ, GW, ML, MR,
             NE, SN, TD, TG
PRIORITY APPLN. INFO.:                    US 2001-348205P  P  20011109
                                          EP 2002-77923    A  20020718
OTHER SOURCE(S):          CASREACT 138:369025; MARPAT 138:369025
AB   Bidentate ligands, R1R2M1-R-M2R3R4, (wherein M1, M2, independently = P,
     As, Sb; R1, R2, R3, R4, independently = substituted organic group and at
     least one of R1, R2, R3 and R4 contains a tertiary carbon atom through
     which the group is linked to M1 or M2; R = bridging group based on a
     trimethylene group connecting M1 and M2 of which the middle carbon atom is
```

double bonded to a non-metal element chosen from group 14, 15 or 16 of the periodic table of elements) were prepared For example, di-tert-butylphosphine is reacted with 3-chloro-2-chloromethyl-1-propene to give the corresponding bisphosphonium salt, which is treated with NaOH to give 66% 3-(di-tert-butylphosphino)-2-(di-tert-butylphosphinomethyl)-1-propene, (dtbpm). The prepared compds. are ligands in the palladium catalyzed carbonylation of ethylenically or acetylenically unsatd. compds. For example, 3-pentenenitrile undergoes carbonylation in the presence of dtbpm and Pd(OAc)2 to give the corresponding linear 5-cyanovaleric ester.

IT **627-93-0P**
RL: SPN (Synthetic preparation); **PREP (Preparation)**
(preparation of bidentate ligands for use in palladium catalyzed carbonylation of ethylenically or acetylenically unsatd. compds., and subsequent reactivity of products of carbonylation)

RN 627-93-0 CAPLUS
CN Hexanedioic acid, dimethyl ester (9CI) (CA INDEX NAME)



L11 ANSWER 2 OF 4 CAPLUS COPYRIGHT 2004 ACS on STN
ACCESSION NUMBER: 2003:146493 CAPLUS
DOCUMENT NUMBER: 138:187402
TITLE: Method for preparation of C4-6 dicarboxylic acid esters from alkali waste liquid generated in manufacturing **caprolactam**
INVENTOR(S): Chou, Hsien-Chun; Wang, Ke-Shun; Wu, Chung-Ru; Liu, Yao Chung; Liu, Yao-Chung
PATENT ASSIGNEE(S): Chunghuahsing Enterprise Co., Ltd., Taiwan
SOURCE: Jpn. Kokai Tokkyo Koho, 12 pp.
CODEN: JKXXAF
DOCUMENT TYPE: Patent
LANGUAGE: Japanese
FAMILY ACC. NUM. COUNT: 1
PATENT INFORMATION:

| PATENT NO. | KIND | DATE | APPLICATION NO. | DATE |
|---|---|---|---|---|
| JP 2003055303 | A2 | 20030226 | JP 2001-387321 | 20011220 |
| US 2003045750 | A1 | 20030306 | US 2002-47835 | 20020114 |

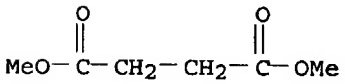
PRIORITY APPLN. INFO.: TW 2001-90119851 A 20010814

AB An efficient process for recovering and manufacturing C4-6 dicarboxylic acids and esters thereof from alkali waste liquid generated in manufacturing **caprolactam** is provided. The process comprises (1) neutralization of the alkali waste liquid generated in manufacturing **caprolactam** with H2SO4 to adjust the pH, separation of the oil layer from the water layer, and addition of HNO3 to the oil layer to convert organic substances into dicarboxylic acids and to obtain a solution of dicarboxylic acids, (2) concentration of the above solution to distill low b.p. monocarboxylic acids and HNO3 in the first stage and concentration and decomposition of residual HNO3 and nitro compds. in the second stage to obtain a crude concentrate mainly containing C4-6 dicarboxylic acids, (3) two-step esterification involving addition of C1-4 alkyl alcs. to the crude C4-6 dicarboxylic acid concentrate followed by obtaining a semiesterification intermediate product in the first stage and the obtaining a crude dicarboxylic acid ester product in the second stage, and (4) distillation of the crude dicarboxylic acid ester product to obtain a single or a mixture of dicarboxylic acid esters. Thus, an oil layer (120 g) containing formic acid 2.08, acetic acid 2.07, butyric acid 3.19, valeric acid 0.23, caproic acid 3.19, succinic acid 0.08, glutaric acid 0.85, adipic acid 10.15, 6-hydroxyhexanoic acid 15.07, H2O 25.62, and others 35.24 weight% (obtained as described in step 1) was added portionwise at 30° over apprx.20 min to 600 g 30% aqueous HNO3, allowed to react at 30° for 30 min, 50° for 1 h, and 70° for 1 h, and concentrated until the temperature reached at 120°, followed by passing steam to drive out HNO3 from the solution, stopping steam, and concentration of the resulting solution at

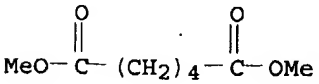
140° to obtain a concentrate (61.5 g) containing formic acid 0, acetic acid 0, butyric acid 0, valeric acid 0.29, caproic acid 0, succinic acid 14.27, glutaric acid 27.36, adipic acid 34.03, 6-hydroxyhexanoic acid 0.53, H2O 0, NO3- 0.045, and others 36.24 weight%. MeOH 100, the crude concentrate 50, and p-toluenesulfonic acid monohydrate 0.5 g were added to a glass flask fitted with a reflux condenser and allowed to react at 80° for 2 h for the first-stage esterification, followed by removing the reflux condenser, distilling residual MeOH and water formed, raising the temperature to 110°, gradually adding 200 g MeOH over 4 h for the second-stage esterification, and distilling unreacted MeOH and H2O formed to give crude dicarboxylic acid ester product (53.5 g) containing succinic acid Me ester 14.13, glutaric acid Me ester 27.68, and adipic acid Me ester 32.64 weight%. The crude dicarboxylic acid ester product (50 g) was distilled at 4 Kpa (130 Torr) to collect the distillate at 110-190°. A clear mixture of C4-6 dicarboxylic acid Me ester (35.1 g) of high purity containing succinic acid Me ester 17.93, glutaric acid Me ester 37.58, and adipic acid Me ester 41.61 weight% was obtained.

IT **106-65-0P**, Succinic acid dimethyl ester **627-93-0P**, Adipic acid dimethyl ester **1119-40-0P**, Glutaric acid dimethyl ester
RL: IMF (Industrial manufacture); SPN (Synthetic preparation); **PREP (Preparation)**
(preparation of C4-6 dicarboxylic acid esters from alkali waste liquid generated in manufacturing **caprolactam** by neutralization, oxidation with nitric acid, concentration, two-stage esterification with alkanols, and distillation)

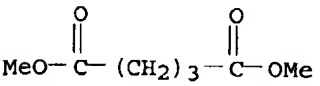
RN 106-65-0 CAPLUS
CN Butanedioic acid, dimethyl ester (9CI) (CA INDEX NAME)



RN 627-93-0 CAPLUS
CN Hexanedioic acid, dimethyl ester (9CI) (CA INDEX NAME)



RN 1119-40-0 CAPLUS
CN Pentanedioic acid, dimethyl ester (9CI) (CA INDEX NAME)



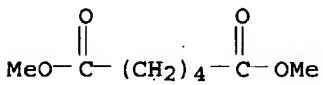
L11 ANSWER 3 OF 4 CAPLUS COPYRIGHT 2004 ACS on STN
ACCESSION NUMBER: 1976:43400 CAPLUS
DOCUMENT NUMBER: 84:43400
TITLE: **Caprolactam** technology. Thermal oxidation of cyclohexanone
AUTHOR(S): Stanek, J.; Capek, A.; Ledvinova, M.
CORPORATE SOURCE: Chemopet. Koncernovy Podnik Spolana, Neratovice, Czech.
SOURCE: Polyamidy '75, Sb. Prednasek (1975), 24-30. Dum Tech. CVTS: Pardubice, Czech.
CODEN: 31WAAO
DOCUMENT TYPE: Conference
LANGUAGE: Czech

AB Acid impurities in tech. cyclohexanone (I) and its oxidation products by air at 100° were determined as Me esters by gas liquid chromatog. and mass

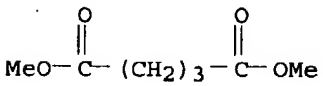
spectra. Me esters and their relative concns. (%) are given for tech. I and its oxidation products: valerate (2.1, 12.6), caproate (9.7, 14.1), 5-hexenoate/4-hexenoate (8.9, 1.9), glutarate (5.5, 13.5), adipate (50, 46.3), and 6-hydroxycaproate (5.8, -). An oxidation scheme was proposed.

IT **627-93-0P 1119-40-0P**
RL: **PREP (Preparation)**
(by oxidation of cyclohexanone)

RN 627-93-0 CAPLUS
CN Hexanedioic acid, dimethyl ester (9CI) (CA INDEX NAME)



RN 1119-40-0 CAPLUS
CN Pentanedioic acid, dimethyl ester (9CI) (CA INDEX NAME)

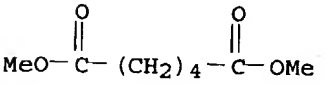


L11 ANSWER 4 OF 4 CAPLUS COPYRIGHT 2004 ACS on STN
ACCESSION NUMBER: 1971:463062 CAPLUS
DOCUMENT NUMBER: 75:63062
TITLE: Reprocessing of an aqueous layer, formed during the production of **caprolactam**, into dimethyl esters of dicarboxylic acids
AUTHOR(S): Freidlin, G. N.; Golubko, L. A.; Glushkova, A. A.; Enyutina, T. M.; Romanova, L. G.; Sapol'kova, G. K.; Samoshina, T. F.; Boyarkin, M. A.
CORPORATE SOURCE: USSR
SOURCE: Khimicheskaya Promyshlennost (Moscow, Russian Federation) (1971), 47(6), 431-3
CODEN: KPRMAW; ISSN: 0023-110X
DOCUMENT TYPE: Journal
LANGUAGE: Russian

AB The title aqueous layer, containing adipic 21, glutaric 5, and 2 oxalic acids 3%, and smaller amts. of cyclohexanol. cyclohexanone, esters and tar, was oxidized at 2.5 atm in 2 stages at 78-100° with HNO3. The H2O and HNO3 were distilled in vacuo to give a melt containing 1.5-3.0% HNO3 and 97% dicarboxylic acids comprising adipic 19-24, glutaric 42-55, succinic 25-33, and oxalic 0.2-4%. The melt was converted to Me esters at 200° with MeOH in 2 hr in an autoclave. Distillation in vacuo gave the Me esters in 92-4% purity, suitable for plasticizer use.

IT **627-93-0P 1119-40-0P**
RL: **PREP (Preparation)**
(recovery of, in **caprolactam** manufacture)

RN 627-93-0 CAPLUS
CN Hexanedioic acid, dimethyl ester (9CI) (CA INDEX NAME)



RN 1119-40-0 CAPLUS
CN Pentanedioic acid, dimethyl ester (9CI) (CA INDEX NAME)